

(19)



(11)

EP 1 642 988 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:
20.07.2011 Bulletin 2011/29

(51) Int Cl.:
C21D 8/06 ^(2006.01) **B21B 1/18** ^(2006.01)
C21D 8/00 ^(2006.01)

(21) Application number: **04734146.6**

(86) International application number:
PCT/JP2004/007277

(22) Date of filing: **20.05.2004**

(87) International publication number:
WO 2004/104235 (02.12.2004 Gazette 2004/49)

(54) **WARM ROLLING METHOD**

WARMWALZVERFAHREN

PROCEDE DE LAMINAGE A CHAUD

(84) Designated Contracting States:
DE FR GB IT

(30) Priority: **20.05.2003 JP 2003180290**

(43) Date of publication of application:
05.04.2006 Bulletin 2006/14

(73) Proprietor: **National Institute for Materials Science**
Tsukuba-shi,
Ibaraki 305-0047 (JP)

(72) Inventors:

- **TORIZUKA, Shiro,**
c/o Nat. Inst. for Materials Sci.
Tsukuba-shi,
Ibaraki 305-0047 (JP)
- **MURAMATSU, Eijiro,**
c/o Nat. Inst. for Materials Sc
Tsukuba-shi,
Ibaraki 305-0047 (JP)
- **INOUE, Tadanobu,**
c/o Nat. Inst. for Materials Sci.
Tsukuba-shi,
Ibaraki 305-0047 (JP)
- **NAGAI, Kotobu,**
c/o Nat. Inst. for Materials Sci.
Tsukuba-shi,
Ibaraki 305-0047 (JP)

(74) Representative: **Calamita, Roberto**
Dehns
St Bride's House
10 Salisbury Square
London
EC4Y 8JD (GB)

(56) References cited:

EP-A- 1 559 804	WO-A-02/20189
DE-A1- 3 545 952	JP-A- 2000 309 823
JP-A- 2000 309 850	JP-A- 2002 137 002
JP-A- 2002 192 201	JP-A- 2003 253 332
US-A- 5 325 697	US-B1- 6 205 835

- **PATENT ABSTRACTS OF JAPAN** vol. 2000, no. 13, 5 February 2001 (2001-02-05) -& JP 2000 301204 A (DAIDO STEEL CO LTD), 31 October 2000 (2000-10-31)
- **PATENT ABSTRACTS OF JAPAN** vol. 007, no. 092 (M-208), 16 April 1983 (1983-04-16) -& JP 58 016704 A (SUMITOMO KINZOKU KOGYO KK), 31 January 1983 (1983-01-31)
- **OHMORI AKIO ET AL.:** 'Mechanical properties of warm rolled steel plates with ultrafine-grained ferrite and cementite structures' **CURRENT ADVANCES IN MATERIALS AND PROCESSES** vol. 14, 2001, page 1050, XP002983045
- **OMORI ET AL.:** 'Onkan ta-pass-ko roll atsuen ni yoru chobisai ferrite soshikiko no sosei' **TETSU TO HAGANE** vol. 89, no. 7, 01 July 2003, pages 781 - 788, XP002983046

Note: Within nine months of the publication of the mention of the grant of the European patent in the European Patent Bulletin, any person may give notice to the European Patent Office of opposition to that patent, in accordance with the Implementing Regulations. Notice of opposition shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

EP 1 642 988 B1

Description

[0001] The present invention relates to a new warm rolling method for manufacturing an ultrafine grain steel material with an ultrafine grain structure of 3 μm or smaller in grain size and excellent in strength and ductility.

[0002] An ultrafine grain steel material can be extremely enhanced in strength without adding alloying elements, and is considered to be decreased extremely in the ductile to brittle transition temperature at the same time, and hence the present inventors have been promoting researches in order to realize the ultrafine grain steel industrially, and have so far invented a method of warm multi-pass rolling (document 1) and a method of multi-directional working (document 2).

[0003] If warm multi-directional rolling can be realized easily, it may lead to wider use of ultrafine grain steel, but it was found not easy in the process of studies by the inventors.

[0004] As one of the technical difficulties, not less than a certain amount of strain must be introduced into the material. For example, the critical strain is 1.5 to 2.3, preferably about 3, and a strain of 3 corresponds to a section reduction ratio of 95%, and a large deformation processing is needed. To obtain a round bar of 10 mm in diameter as final product, warm rolling process must be started from diameter of 45 mm, and to introduce such large strain in warm rolling temperature region of high deformation resistance, a large material is needed. As a result, the number of rolling passes increases.

[0005] If a larger strain can be introduced into the material by a smaller section reduction ratio and a smaller number of passes, an ultrafine grain structure can be obtained more easily, lots of industrial benefits are expected such as enhancement of rolling efficiency.

[0006] The inventors have proposed various methods about multi-directional rolling, such as a method of compressing from multiple directions by using anvil (document 2) and two-directional screw-down rolling technology. Although the multi-directional working is a technology for introducing a large amount of strain efficiently, but processing from at least two directions involved very difficult technical problems.

Document 1: Japanese Patent Application Laid-Open No. 2000-309850

Document 2: Japanese Patent Application Laid-Open No. 2001-240912

[0007] The present invention is devised in the light of the above background, and is intended to present a new multi-directional warm rolling method capable of introducing a large strain into the material by a smaller section reduction ratio and a smaller number of passes, by easier means, by further advancing from the findings obtained so far by the inventors, and a manufacturing method of steel material having ultrafine grain structure, and excellent in strength and ductility.

[0008] To solve the problems, in a first aspect the invention provides a warm rolling method for manufacturing an ultrafine grain steel material having an ultrafine grain structure of average grain size of 3 μm or less, which method comprises applying, at least once, rolling by an oval shaped caliber followed by rolling a square shaped caliber, in a rolling temperature range of 350°C to 800°C, to a steel material which has a chemical composition, by wt%, of

C: 0.001% or more to 1.2% or less;

Si: 0.1% or more to 2% or less;

Mn: 0.1% or more to 3% or less;

P: 0.2% or less;

S: 0.2% or less;

Al: 1.0% or less;

N: 0.02% or less;

optionally, Cr, Mo, Cu and Ni in total: 30% or less;

optionally, Nb, Ti, V in total: 0.5% or less;

optionally, B: 0.01% or less

balance of Fe and inevitable impurities,

wherein after the rolling by an oval shaped caliber, the length of the shortest cross-sectional axis of said material is 75% or less of the material diagonal length before said rolling by oval shaped caliber.

[0009] Preferably the rolling by oval shape caliber is executed by 2 times or more to maximum N/2 times or less in the case of N>2, wherein N is the total number of passes. Two continuous rolling passes may be executed. The section reduction ratio after square shape caliber rolling from material may be 20% or more in two passes rolling by calibers of oval shape and square shape. The section reduction ratio may be 40% or more in combination rolling by two times, and the section reduction ratio may be 60% or more in combination rolling by three times, in combination rolling of two passes rolling by calibers of oval shape and square shape.

[0010] A plastic strain of 1.5 or more may be introduced at least in a region of 50 vol.% inside of the material. More preferably, a plastic strain of 2 or more may be introduced in a region of 90 vol.% or more inside of the material. In a

second aspect the invention provides a warm rolling method in which the rolling condition parameter Z expressed in the following formula (1) is 11 or more (the structure just before rolling is ferrite, bainite, martensite, pearlite or other Fe crystal structure of bcc) or 20 or more (structure just before rolling is austenite and Fe crystal structure of fcc).

$$Z = \log \left[\frac{\varepsilon}{t} \exp \left(\frac{Q}{8.31(T + 273)} \right) \right] \quad (1)$$

ε: strain

τ: time from start of rolling till end (s)

T: rolling temperature (°C, average of rolling temperature of each pass in the case of multi-pass rolling)

Q: 254,000 if structure just before rolling is a primary phase of ferrite, bainite, martensite or pearlite; or 300,00 if mother phase is austenite.

[0011] The invention having such features is devised on the basis of new findings obtained by the investigations by the inventors. That is, hitherto, it is known that caliber rolling which is rolling by using a roll having a hole groove is common as the steel bar manufacturing method, and shapes of caliber are roughly classified into square shape (square shape, diamond shape), oval shape and round shape. By performing caliber (groove roll) rolling in warm rolling temperature region, a structure mainly composed of ultrafine grain ferrite is obtained by multi-pass rolling (document 1). By using the oval shape caliber, it is found to be effective for equiaxialization of ferrite grain shape of L section of steel bar (section parallel to longitudinal direction of bar).

[0012] As a result of intensive studies by the inventors, it has been found that a large strain can be introduced into the material even at a relatively small section reduction ratio, by performing caliber rolling combining oval shape caliber and square, round or other shape caliber, in an appropriate temperature region, so that the technology is established.

[0013] In the accompanying drawings :

Fig. 1 is a diagram of caliber in embodiment 1.

Fig. 2 is a photograph of C section of steel bar after rolling.

Fig. 3 is a material mesh diagram.

Fig. 4 is a diagram showing plastic strain after 1 pass of oval shape caliber rolling.

Fig. 5 is a diagram showing plastic strain after 2 passes of square shape caliber rolling.

Fig. 6 is a diagram showing plastic strain after 3 passes of oval shape caliber rolling.

Fig. 7 is a diagram showing plastic strain after 4 passes of square shape caliber rolling.

Fig. 8 is a diagram showing plastic strain after 5 passes of oval shape caliber rolling.

Fig. 9 is a diagram showing plastic strain after 6 passes of round shape caliber rolling.

Fig. 10 is a SEM image of structure after 2 passes of square shape caliber rolling.

Fig. 11 is a SEM image of structure after 4 passes of square shape caliber rolling.

Fig. 12 is a SEM image of structure in embodiments 2 to 4.

Fig. 13 is a diagram of caliber.

Fig. 14 is a photograph of C section of steel bar after rolling.

Fig. 15 is a SEM image of structure.

Fig. 16 is a SEM image of structure of comparative example 1.

Fig. 17 is a diagram showing the relation of parameter Z and average grain size.

[0014] The invention has the features as mentioned above, and the individual embodiments are described specifically below.

[0015] The warm rolling method of the invention is capable of manufacturing an ultrafine grain steel material having an ultrafine grain structure of average grain size of 3 μm or less, as mentioned above, by combining rolling by a caliber of oval shape and rolling by a caliber of other shape. In this case, the caliber rolls used in rolling are oval shape caliber and other shape caliber.

[0016] As for the caliber roll of oval shape caliber, the hole shape formed by upper die and lower die is not circular (round), but the shape that circular (round) is flattened. The caliber of other shape combined with the oval shape caliber includes square, rhombus (diamond), round or similar shapes thereof.

[0017] In the invention, as the warm rolling method for manufacturing an ultrafine grain steel material having an ultrafine grain structure of average grain size of 3 μm or less, both of rolling by oval shape caliber and rolling by other shape caliber are executed at least once or more in rolling of two passes or more to steel material in rolling temperature range

from 350 to 800°C.

[0018] Actually, a preferable embodiment is that rolling by a caliber of oval shape is followed by rolling by a caliber of other shape, and that the rolling by oval shape caliber is executed by 2 times or more to maximum N/2 times or less in the case of N>2, wherein N is the total number of passes.

[0019] For example, when combining oval shape caliber and square caliber, the rolling condition is considered so as to include, in all passes of rolling, two times or more of combination rolling using oval shape caliber and square shape caliber (oval-square), to include rolling by square shape caliber in the middle of combination rolling of oval-square such as oval-square-square-oval-square, or to roll of 4 passes of oval-square-oval-square, or to roll of 6 passes of oval-square-oval-square-oval-square. In this case, too, the square shape caliber may be replaced by a caliber of round, rhombus or other shape.

[0020] In the rolling method of the invention, a microscopic local orientation difference caused by introduction of large strain by warm rolling originates ultrafine grain, and in the recovery process taking place during or after working, the dislocation density in grain decreases and grain boundary is formed at the same time, and thereby an ultrafine grain structure is formed. However, the recovery is not sufficient if the temperature is low, then deformation texture with high dislocation density is remained. On the other hand, if the temperature is too high, the grain becomes coarse due to discontinuous recrystallization or ordinary grain growth, and ultrafine grain structure of 3 μm or less is not formed. Hence, the rolling temperature is limited within 350 to 800°C.

[0021] In the present invention, ultrafine grains are generated from the deformed grains flattened by warm rolling, and increase along with increase of strain. And, a strain of at least 1.5 is required in order to obtain a structure almost composed of ultrafine grains.

[0022] More specifically, by introducing a plastic strain of 1.5 or more, preferably 2 or more, in the region of at least 50 vol.% of the material inside, ultrafine grains can be formed in this region. Preferably, by introducing a plastic strain of 2 or more in the region of 90 % or more of material inside, an ultrafine grain region can be formed in this region.

[0023] The greater the strain to be introduced is, the greater the orientation differential angle among ultrafine grains is. That is, large angular grain boundaries increase. If a strain of 3 can be introduced, the rate of large angular grain boundaries is sufficient in the grain boundary of ultrafine grains. Therefore, as far as the region of strain of 3 or more is 50% or more, or preferably 80% or more of the entire section, a steel bar of excellent dynamic properties is obtained.

[0024] In addition to processing in principal screw-down direction, when combined with screw-down from other direction forming an angle of about 90°, by applying processing strain from at least two directions, the orientation of ultrafine grains is dispersed, and the rate of large angular grain boundaries can be increased.

[0025] According to the studies accumulated so far by the inventors, it has been disclosed that the average grain size of ultrafine grains formed by warm rolling depends on the processing temperature and strain speed. The grain size becomes smaller along with increase of rolling condition parameter Z of formula (1) as the function of rolling temperature and strain speed. To obtain a structure of average grain size of 1 μm or less, the rolling condition parameter Z must be set higher than a specific critical value. As a result of experiment by one-pass large strain compressive working using small samples, the critical value is found to be about 11 in the case of bcc structure iron (ferrite, bainite, martensite, pearlite, etc.), and about 20 in fcc structure (austenite) (Fig. 17).

[0026] In formula (1), strain (ε) may be a true strain that is industrially easy-to-use strain. For example, supposing the initial area of steel bar to be S₀, and the area of C section after rolling to be S, the section reduction ratio R is

$$R = (S_0 - S) / S_0 \quad (2)$$

Hence, the true strain ε is expressed as follows;

$$\epsilon = -\ln (1-R)$$

[0027] Instead of true strain, the value calculated by finite element method may be used (for example, Keizaburo Harumi, et al. "Introduction to finite element method," Kyoritsu Publishing, March 15, 1990). More specifically, the plastic strain is calculated according to the flow shown in Table 1 below.

Table 1

Calculation flow of plastic strain

1. Obtain stress-strain curve corresponding to the processing temperature of material.
 2. Prepare for finite element calculation method.
- (1) Create mesh in workpiece.

(continued)

Calculation flow of plastic strain

(2) Determine contact condition: coefficient of friction = 0.3 coulomb condition.

(3) Determine stress-strain curve, material property values.

3. On the basis of conditions of (1) to (3), calculate by universal finite element method, for example, ABAQUS. The plastic strain ε is calculated in the formula below, and each strain increment is calculated by the universal finite element method code.

$$\varepsilon = \frac{2}{3} \sqrt{\left[\frac{1}{2} \left\{ (d\varepsilon_x - d\varepsilon_y)^2 + (d\varepsilon_y - d\varepsilon_z)^2 + (d\varepsilon_z - d\varepsilon_x)^2 \right\} + \frac{3}{4} (d\gamma_{xy}^2 + d\gamma_{yz}^2 + d\gamma_{zx}^2) \right]}$$

 $d\varepsilon_x d\varepsilon_y d\varepsilon_z$: strain increment of x, y, z $d\gamma_{xy} d\gamma_{yz} d\gamma_{zx}$: shearing stress increment

[0028] In the warm rolling method of the invention, hence, it is preferred to set the rolling condition so that parameter Z may be 11 or more (bcc structure) or 20 or more (fcc structure).

[0029] Preferred embodiments of the invention include rolling processes wherein section reduction ratio is 20% or more in two passes rolling of oval shape caliber rolling and square shape caliber rolling to material, section reduction ratio is 40% or more in rolling of combined two times of two passes rolling by oval shape caliber and square shape caliber, section reduction ratio is 60% or more in rolling of combined three times of two passes rolling, and the maximum shorter axis length of material after rolling by oval shape caliber is 70% or less of the diagonal length of material before oval rolling.

[0030] Further, concerning the composition of the steel material to which the warm rolling method of the invention can be applied, the composition of steel is not limited at all because mechanism for heightening the strength by phase transformation is not utilized at all and addition of alloying element is not needed for enhancing the strength, and therefore steel materials of wide composition range can be used such as steel types free from phase transformation, for example, ferrite single phase steel or austenite single phase steel. More specifically, the following composition, by wt.%, is preferred.

C: 0.001% or more to 1.2% or less,

Si: 0.1% or more to 2% or less,

Mn: 0.1% or more to 3% or less,

P: 0.2% or less,

S: 0.2% or less,

Al: 1.0% or less,

N: 0.02% or less,

Cr, Mo, Cu, and Ni in total: 30% or less,

Nb, Ti, V in total: 0.5% or less,

B: 0.01% or less, and

balance of Fe and inevitable impurities. Such composition free from alloying elements may be presented as an example. The alloy elements such as Cr, Mo, Cu, Ni, Nb, Ti, V, B, etc. may be added more than the specified range as required, or may not be added at all.

[0031] Presenting embodiments, the invention is more specifically described below. But it must be noted that the invention is not limited to the embodiments alone.

Embodiments

[0032] Table 2 shows chemical composition of sample steels used in embodiments (the balance is Fe).

Table 2

Chemical composition of sample steels (mass %)

	C	Si	Mn	P	S	Al
a	0.15	0.3	1.5	0.01	0.001	0.03

(continued)

	C	Si	Mn	P	S	Al
b	0.11	0.3	0.5	0.02	0.005	0.03

<Embodiment 1>

[0033] A steel bar of 24 mm square having a ferrite + pearlite structure of average ferrite grain size of 5 microns of the composition shown in Table 2a was rolled in 6-pass caliber using the caliber shown in Fig. 1 at rolling temperature of 520 to 450°C. In Fig. 1, the outline of caliber dimension (mm) is as shown in Table 3.

Table 3

	Longer axis	Shorter axis	Radius of curvature
1st pass, oval	54	12	64
3rd pass, oval	41	9	49
5th pass, oval	19	10	12
6th pass, round		Diameter	12

[0034] Fig. 2 shows the sectional shape changes and section reduction ratio of each pass of rolling. The section reduction ratio of rolling the 24 x 24 mm square bar by oval shape caliber in the first pass is 37%, the section reduction ratio of rolling material by square shape caliber in the second pass is 21%, the section reduction ratio of rolling material by oval shape caliber in the third pass is 15%, the section reduction ratio of rolling material by square shape caliber in the fourth pass is 24%, the section reduction ratio of rolling material by oval shape caliber in the fifth pass is 13%, and the section reduction ratio of rolling material by round shape caliber in the sixth pass is 12%. The section reduction ratio from the material to the square bar of 17 mm in the second pass is 44%, the section reduction ratio from the material to the square bar of 13 mm in the fourth pass is 71%, and the section reduction ratio from the material to the round bar of 12.5 mm in the sixth pass is 80%.

[0035] Fig. 3 to Fig. 9 show distribution of plastic strain in the material inside calculated by the finite element method. Fig. 5 suggests there is a region exceeding the plastic strain of 1.5 in the material already in the second pass of oval-square shape caliber. Its area rate is 75%. As shown in Fig. 6, after rolling of three passes of oval-square-oval, a region over plastic strain of 2 occupies 92% of all area, and in Fig. 7, after rolling of four passes of oval-square-oval-square, a region over plastic strain of 3 occupies 95%, and after oval-round rolling in Fig. 9, the plastic strain is 3 or more in 100% region.

[0036] The section reduction ratio after two passes is about 44% (when section reduction ratio R is converted merely into true strain ϵ , from $\epsilon = -\ln(1-R/100)$, $\epsilon = 0.67$), after four passes, 71% (section reduction R merely converted into true strain of 1.23), and after six passes, 80% (section reduction R merely converted into true strain of 1.61), but it is found that a very large plastic strain is formed inside the material. This is because the oval shape caliber and square shape caliber are combined in rolling, and the strain is far larger than the strain calculated from a mere section reduction area.

[0037] Fig. 10 and Fig. 11 show SEM images of the structure. In positions ① and ② in Fig. 10 corresponding to Fig. 5, ultrafine ferrite grains of 1 micron or less are produced, while ultrafine grains are not found at position of ③. In the microstructure of Fig. 11 corresponding to Fig. 7, almost entire region is covered with ultrafine grain structure of ultrafine ferrite grains of 1 micron or less.

[0038] Table 4 shows dynamic properties of material of 13 mm square after four passes. Properties of 24 square bar before rolling are shown for reference. Without causing brittle breakdown at double yield strength and liquid nitrogen temperature, an absorption energy of J was recognized.

Table 4

	Ferrite grain size (μm)	Yield strength (MPa)	Tensile strength (MPa)	Ductile-brittle transition temperature	Absorption energy (J) -120°C	Central Vickers hardness
Embodiment 1	0.5	840	850	<-196	118	290

(continued)

	Ferrite grain size (μm)	Yield strength (MPa)	Tensile strength (MPa)	Ductile- brittle transition temperature	Absorption energy (J) -120°C	Central Vickers hardness
Embodiment 4	0.6	800	810	<-196	80	270-310
Comparative example 2	5	460	580	-40	0	

<Embodiment 2 to 4>

[0039] A steel bar of 24 mm square having a ferrite + pearlite structure of average ferrite grain size of 5 microns of the composition shown in Table 2a was rolled in 2-pass caliber using the caliber shown in Fig. 1 (1), (2) at rolling temperature of 400°C, 600°C, and 700°C. Fig. 12 (a), (b), (c) show SEM images of central part of steel bar (corresponding to position ① in Fig. 10), in which fine ferrite grains of average grain size of 0.5, 1, and 1.5 microns are formed.

<Embodiment 5>

[0040] A steel bar of 15 mm square having a ferrite + pearlite structure of average ferrite grain size of 20 microns of the composition shown in Table 2b was rolled in 6-pass caliber until diameter of 8 mm, using the caliber shown in Fig. 13 at rolling temperature of 450 to 550°C. Table 5 shows outline of caliber dimension. Fig. 14 shows the sectional shape changes and section reduction ratio in each pass of rolling. Fig. 15 shows SEM images of structure after six passes, in which a fine ferrite grain structure was formed in spite of section reduction ratio of about 74%. Concerning dynamic properties, the Vickers hardness is shown in the bottom of the photograph in Fig. 15, and an excellent property of over 800 MPa is obtained at tensile strength of 270 to 310.

Table 5

	Longer axis	Shorter axis	Radius of curvature
1st pass, oval	31	6.8	38
3rd pass, oval	27	5.3	35.9
5th pass, oval	15	6.5	10.7
6th pass, round		Diameter	8

<Comparative example 1>

[0041] A steel bar of 24 mm square having a ferrite + pearlite structure of average ferrite grain size of 5 microns of the composition shown in Table 2a was rolled in 7-pass caliber at section reduction ratio of 70% (strain 1.2) until 13 mm square, using the caliber shown in Fig. 1 at rolling temperature of 500°C. It was not rolled by oval shape caliber. As shown in SEM image in Fig. 16, fine grains were not formed in the center of the steel bar.

<Comparative example 2>

[0042] A steel bar of 115 mm square of the composition shown in Table 2a was heated to 900°C, and rolled in caliber at section reduction ratio of 94% (strain 3.1) until 24 mm square, using the square shape caliber at rolling temperature of 870 to 850°C. It was not rolled by oval shape caliber. The average grain size was 5 μm , and fine grains were not formed. Dynamic properties were shown in Table 4, and the yield strength and tensile strength were respectively 460 and 580 MPa.

[0043] As described herein, the invention presents a new warm rolling method capable of introducing a greater strain into the material by a smaller section reduction ratio and a smaller number of passes by an easier means, and further presents a manufacturing method of steel materials excellent in strength and ductility, having ultrafine grain structure.

Claims

1. A warm rolling method for manufacturing an ultrafine grain steel bar material having an ultrafine grain structure of average grain size of 3 μm or less, which method comprises applying, at least once, rolling by an oval shaped caliber followed by rolling by a square shaped caliber, in a rolling temperature range of 350°C to 800°C, to a steel material which has a chemical composition, by wt%, of

C: 0.001 % or more to 1.2% or less;
 Si: 0.1% or more to 2% or less;
 Mn: 0.1 % or more to 3% or less;
 P: 0.2% or less;
 S: 0.2% or less;
 Al: 1.0% or less;
 N: 0.02% or less;

optionally, Cr, Mo, Cu and Ni in total : 30% or less;
 optionally, Nb, Ti, V in total: 0.5% or less;
 optionally, B: 0.01% or less;

balance of Fe and inevitable impurities,

wherein after the rolling by said oval shaped caliber, the length of the shortest cross-sectional axis of said material is 75% or less of the material diagonal length before said rolling by oval shaped caliber.

2. A warm rolling method as claimed in claim 1, wherein rolling is performed based on a rolling condition parameter Z expressed in the following formula (1) is 11 or more in the case that the crystal structure of Fe just before rolling is bcc such as the structure of ferrite, bainite, martensite or pearlite, or 20 or more in the case where the crystalline structure of Fe just before rolling is austenite and fcc;

$$Z = \log \left[\frac{\epsilon}{t} \exp \left(\frac{Q}{8.31(T + 273)} \right) \right] \quad (1)$$

ϵ : strain

t: time from start of rolling till end (s)

T: rolling temperature (°C, average of rolling temperature of each pass in the case of multi-pass rolling)

Q: 254,000 if structure just before rolling is a primary phase of ferrite, bainite, martensite or pearlite; or 300,000 if mother phase is austenite.

Patentansprüche

1. Warmwalzverfahren zur Herstellung eines Ultrafeinkornstahl-Stabmaterials mit einer Ultrafeinkornstruktur mit einer mittleren Korngröße von 3 μm oder weniger, wobei das Verfahren das wenigstens einmalige Anwenden ein Walzens durch eine oval geformte Walzform bzw. Schablone, gefolgt durch ein Walzen durch eine kantig bzw. eckig geformte Walzform in einem Walztemperaturbereich von 350°C bis 800°C auf ein Stahlmaterial umfasst, welches eine chemische Zusammensetzung, in Gew.-%, wie folgt aufweist:

C: 0,001 % oder mehr bis 1,2 % oder weniger;
 Si: 0,1 % oder mehr bis 2 % oder weniger;
 Mn: 0,1 % oder mehr bis 3 % oder weniger;
 P: 0,2 % oder weniger;
 S: 0,2 % oder weniger;
 Al: 1,0 % oder weniger;
 N: 0,02 % oder weniger;

optional Cr, Mo, Cu und Ni, insgesamt: 30 % oder weniger; optional Nb, Ti, V, insgesamt: 0,5 % oder weniger;

optional B: 0,01 % oder weniger;
eine Ausgleichsmenge bzw. ein Rest von Fe und unvermeidbaren Unreinheiten,
wobei nach dem Rollen durch die oval geformte Walzform die Länge der kürzesten Querschnittsachse des Materials
75 % oder weniger der diagonalen Materiallänge vor dem Walzen durch die oval geformte Walzform beträgt.

2. Warmwalzverfahren nach Anspruch 1, wobei das Walzen auf Grundlage eines in der nachfolgenden Gleichung (1) ausgedrückten Walzzustands- bzw. -bedingungsparameters Z durchgeführt wird, der für den Fall, dass die Kristallstruktur von Fe, wie auch die Struktur von Ferrit, Bainit, Martensit oder Pearlit, unmittelbar vor dem Walzen bcc ist, 11 oder mehr beträgt, oder, für den Fall, dass die Kristallstruktur von Fe unmittelbar vor dem Walzen Austenit und fcc ist 20 oder mehr beträgt;

$$Z = \log \left[\frac{\varepsilon}{t} \exp \left(\frac{Q}{8,31(T + 273)} \right) \right] \quad (1)$$

ε : Spannung bzw. Dehnung

t: Zeit vom Beginn des Walzens bis zum Ende (s)

T: Walztemperatur (°C, Mittelwert der Walztemperatur eines jeden Durchgangs für den Fall eines Walzens in mehreren Durchgängen)

Q: 254.000, wenn die Struktur unmittelbar vor dem Walzen eine Primärphase aus Ferrit, Bainit, Martensit oder Pearlit ist; oder 300.000, wenn die Mutterphase Austenit ist.

Revendications

- Procédé de laminage à chaud pour fabriquer un matériau de barre d'acier à grains ultra fins ayant une structure de grain ultra fine de taille de grain moyenne de 3 μm ou moins, lequel procédé comprend l'application, au moins une fois, d'un laminage par un calibre de forme ovale suivie d'un laminage par un calibre de forme carrée, dans une plage de température de laminage de 350 °C à 800 °C, à un acier qui a une composition chimique, en % en poids, de
C : 0,001 % ou plus à 1,2 % ou moins;
Si : 0,1 % ou plus à 2 % ou moins ;
Mn : 0,1 % ou plus à 3 % ou moins ;
P : 0,2 % ou moins ;
S : 0,2 % ou moins ;
Al : 1,0 % ou moins ;
N : 0,02 % ou moins ;
facultativement, Cr, Mo, Cu et Ni au total : 30 % ou moins ;
facultativement, Nb, Ti, V au total : 0,5 % ou moins ;
facultativement, B : 0,01 % ou moins ;
le reste étant Fe et des impuretés inévitables, dans lequel après le laminage par ledit calibre de forme ovale, la longueur du plus petit axe en coupe transversale dudit matériau est de 75 % ou moins de la longueur de diagonale de matériau avant ledit laminage par calibre de forme ovale.
- Procédé de laminage à chaud tel que revendiqué dans la revendication 1, dans lequel le laminage est mis en oeuvre sur la base d'un paramètre de condition de laminage Z exprimé dans la formule (1) suivante qui est 11 ou plus dans le cas où la structure cristalline de Fe juste avant le laminage est cc telle que la structure de la ferrite, de la bainite, de la martensite ou de la perlite, ou 20 ou plus dans le cas où la structure cristalline de Fe juste avant le laminage est austénitique et fcc ;

$$Z = \log \left[\frac{\varepsilon}{t} \exp \left(\frac{Q}{8,31(T + 273)} \right) \right] \quad (1)$$

ε : déformation

t : temps à partir du début du laminage jusqu'à la fin (s)

T : température de laminage (°C, moyenne de la température de laminage de chaque passe dans le cas d'un

EP 1 642 988 B1

laminage à passes multiples)

Q : 254 000 si la structure juste avant le laminage est une phase primaire de ferrite, de bainite, de martensite ou de perlite ; ou 300 000 si la phase mère est austénitique.

5

10

15

20

25

30

35

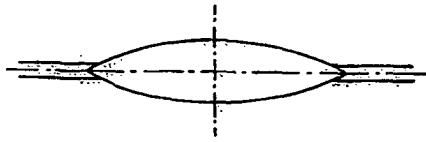
40

45

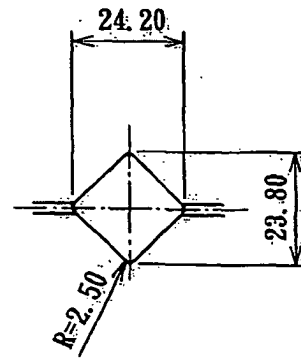
50

55

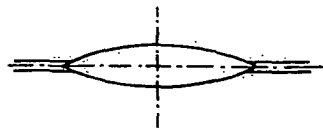
Fig.1



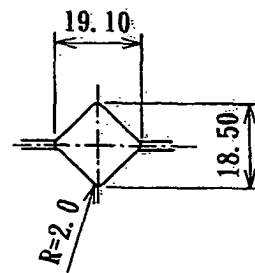
(1) First pass caliber: oval



(2) Second pass caliber: square



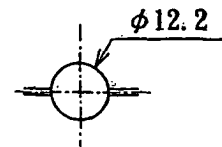
(3) Third pass caliber: oval



(4) Fourth pass caliber: square



(5) Fifth pass caliber: oval



(6) Sixth pass caliber: round

Fig.2

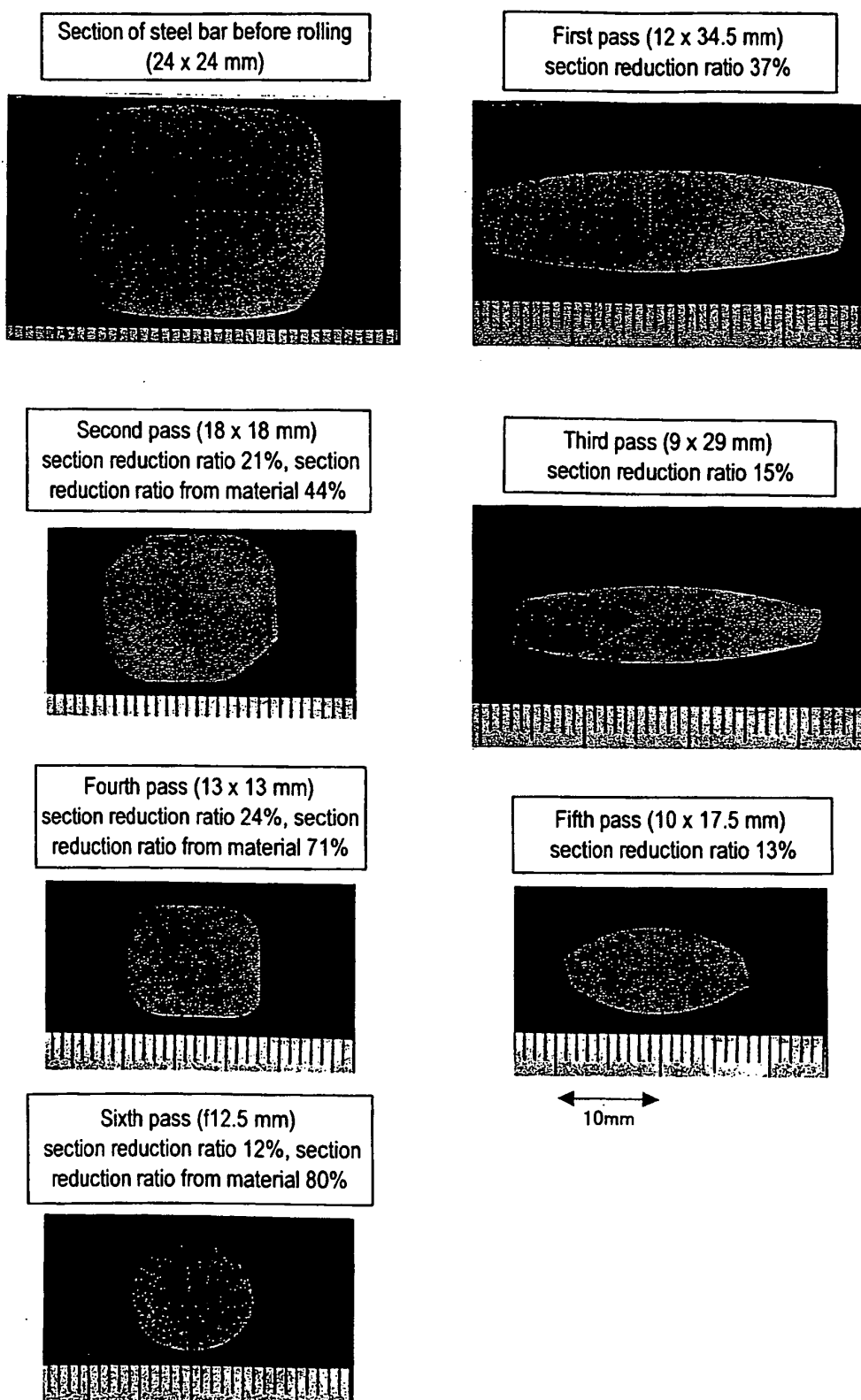


Fig. 3

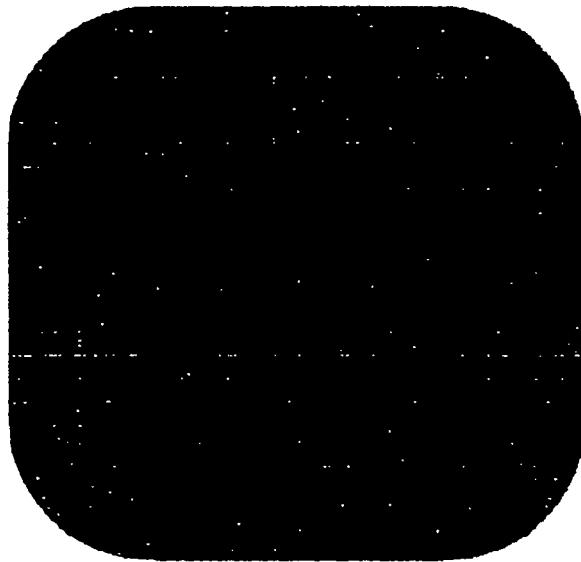


Fig. 4

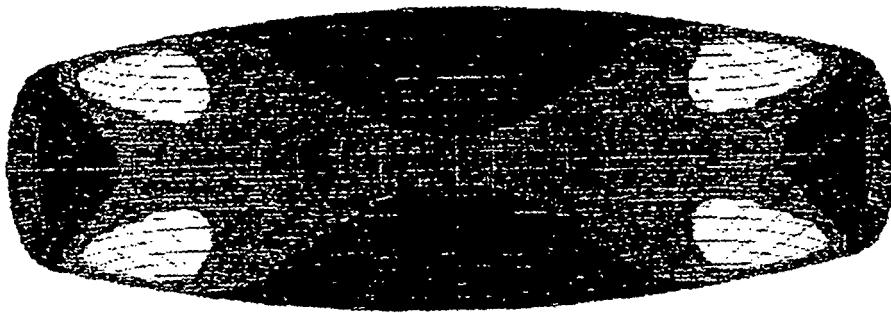


Fig. 5

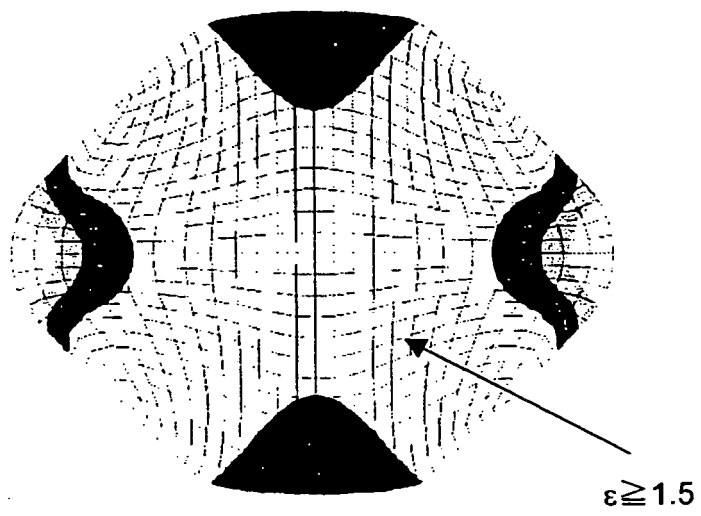


Fig. 6

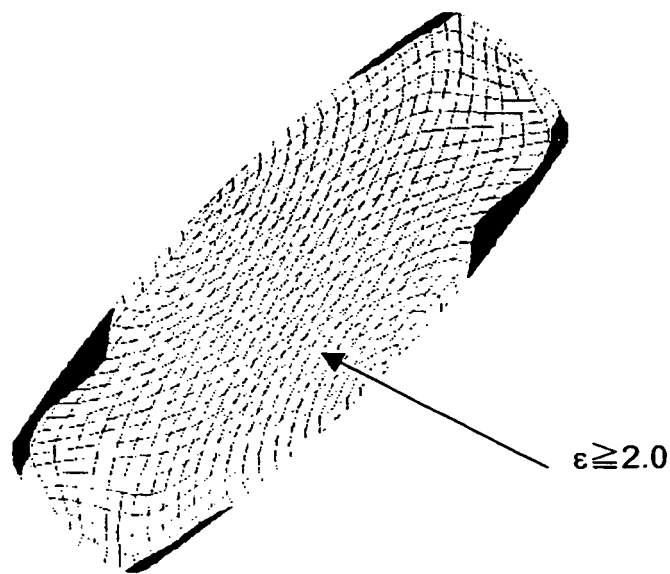


Fig. 7

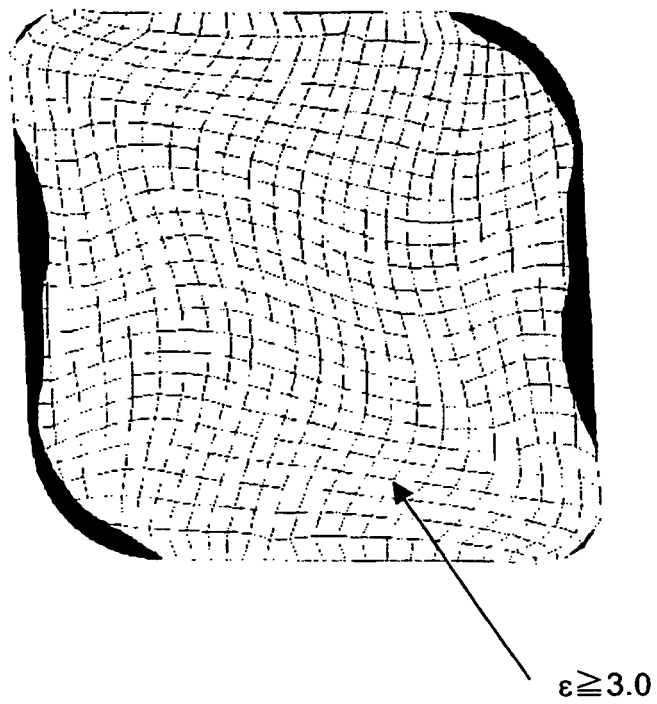


Fig. 8

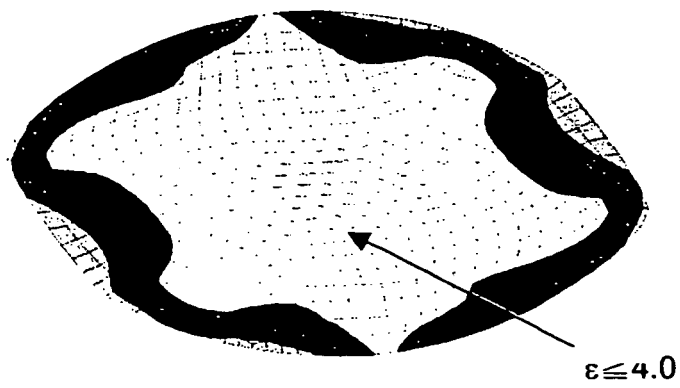


Fig. 9

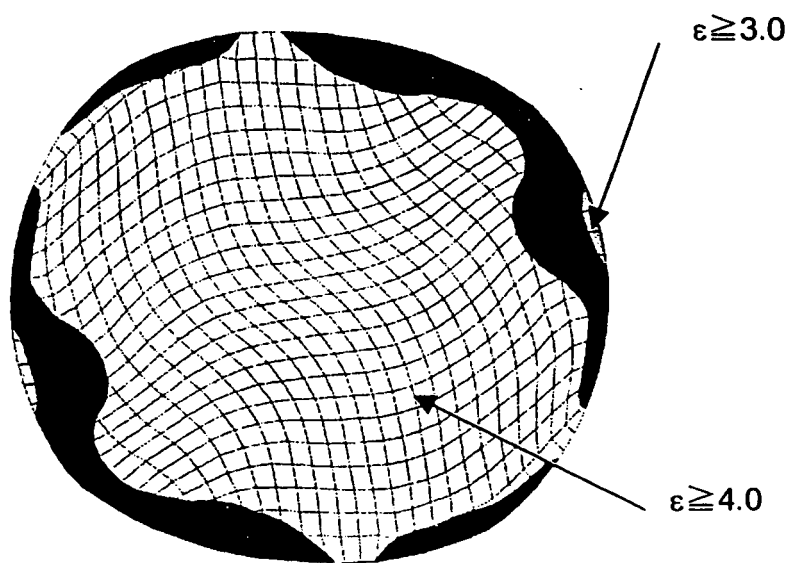


Fig. 10

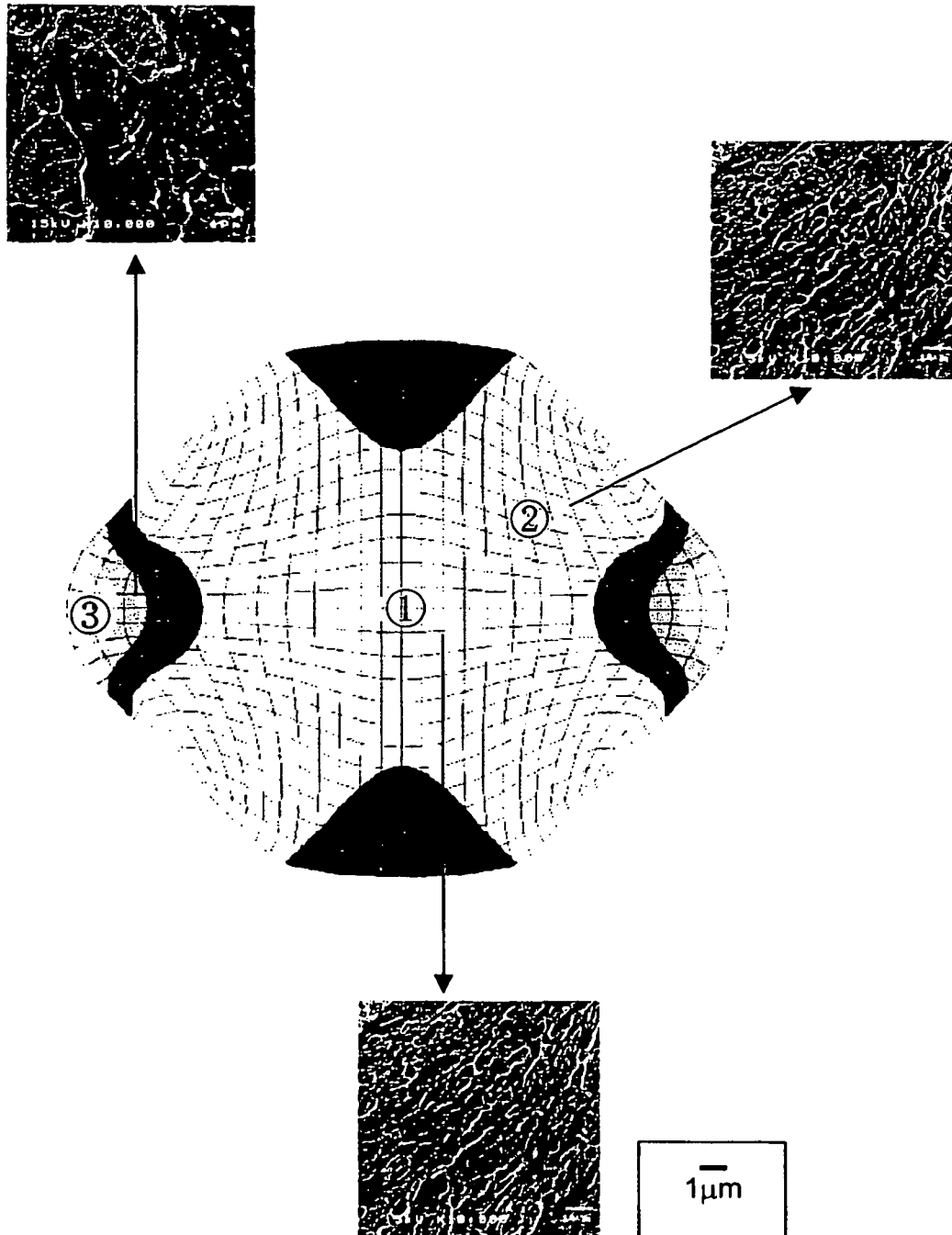


Fig. 11

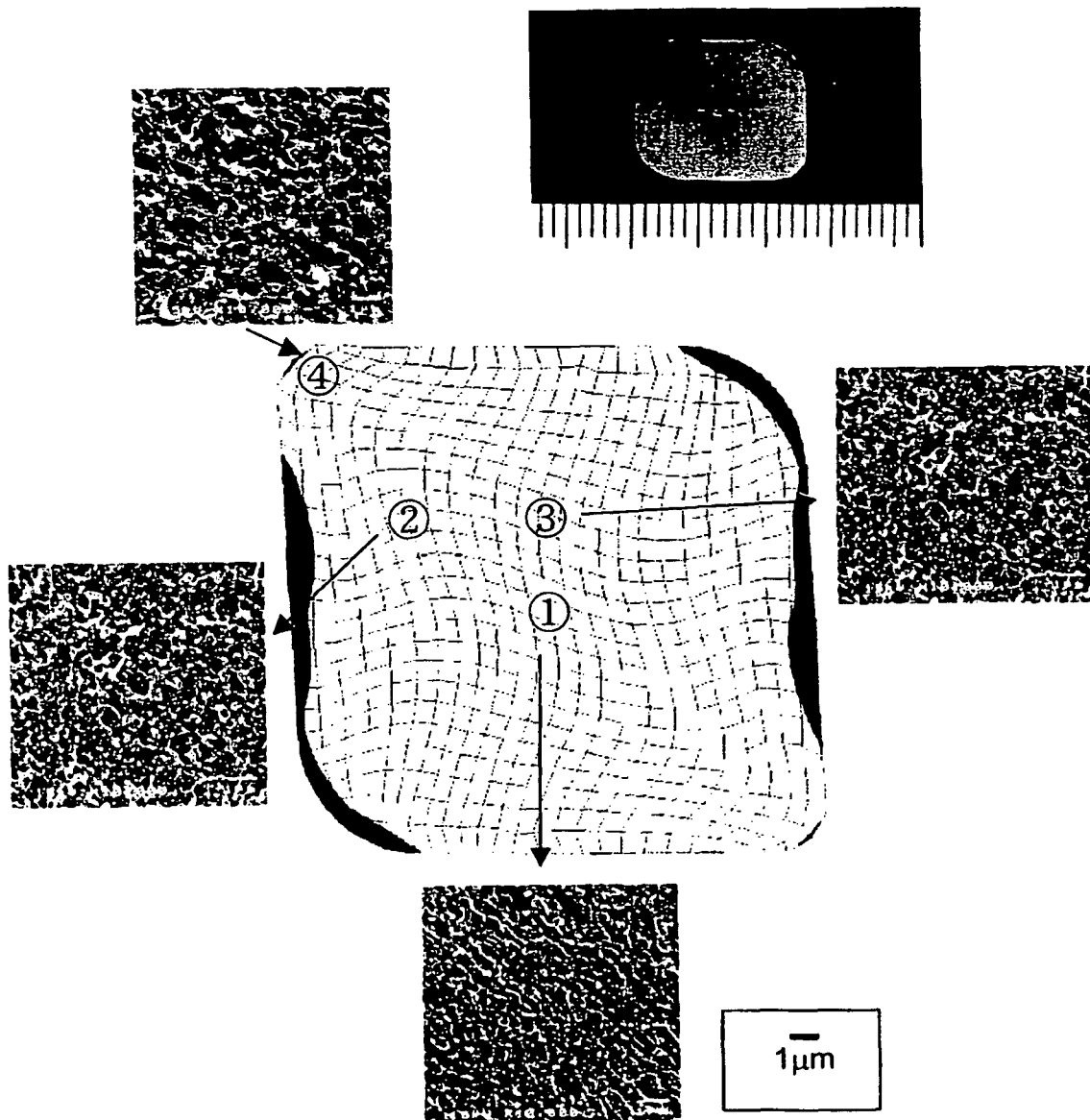
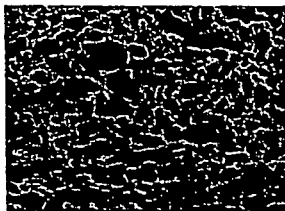
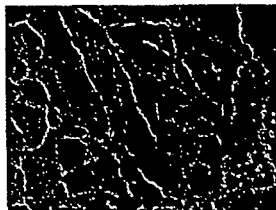


Fig.12

(a) Embodiment 2



(b) Embodiment 3



(c) Embodiment 4

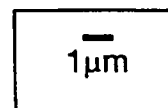
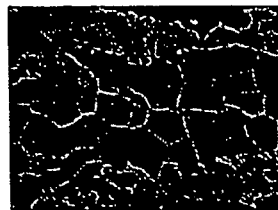
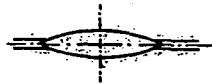
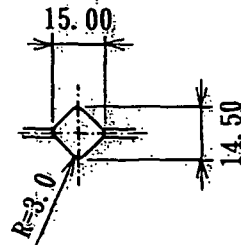


Fig.13



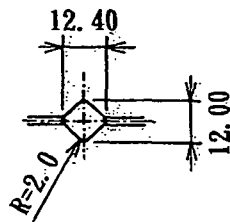
(1) First pass caliber: oval



(2) Second pass caliber: square



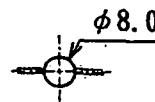
(3) Third pass caliber: oval



(4) Fourth pass caliber: square



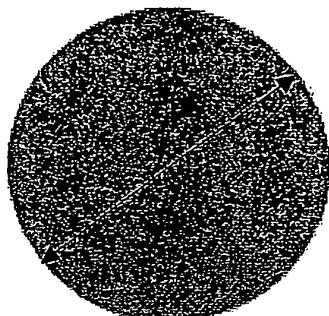
(5) Fifth pass caliber: oval



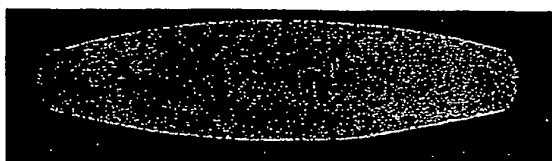
(6) Sixth pass caliber: round

Fig.14

Section of steel bar before rolling
($\phi 15$ mm)



First pass (5.8 x 22.7 mm)
section reduction ratio 39%



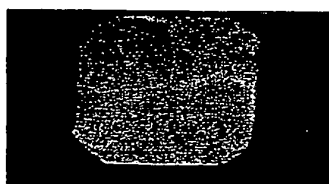
Second pass (9.0 x 10.6 mm)
section reduction ratio 19%, section
reduction ratio from material 51%



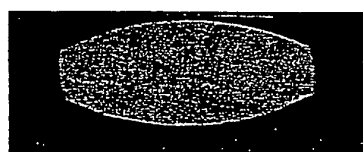
Third pass (4.5 x 17.8 mm)
section reduction ratio 22%



Fourth pass (7.26 x 8.43 mm)
section reduction ratio 17%, section
reduction ratio from material 68%



Fifth pass (5.0 x 11.7 mm)
section reduction ratio 12%



Sixth pass ($\phi 7.8$ mm)
section reduction ratio 10%, section
reduction ratio from material 74%

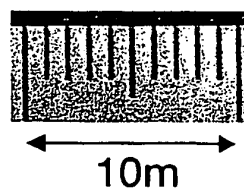
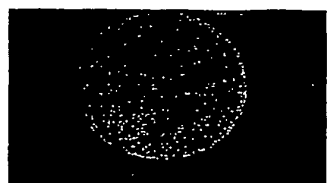


Fig.15

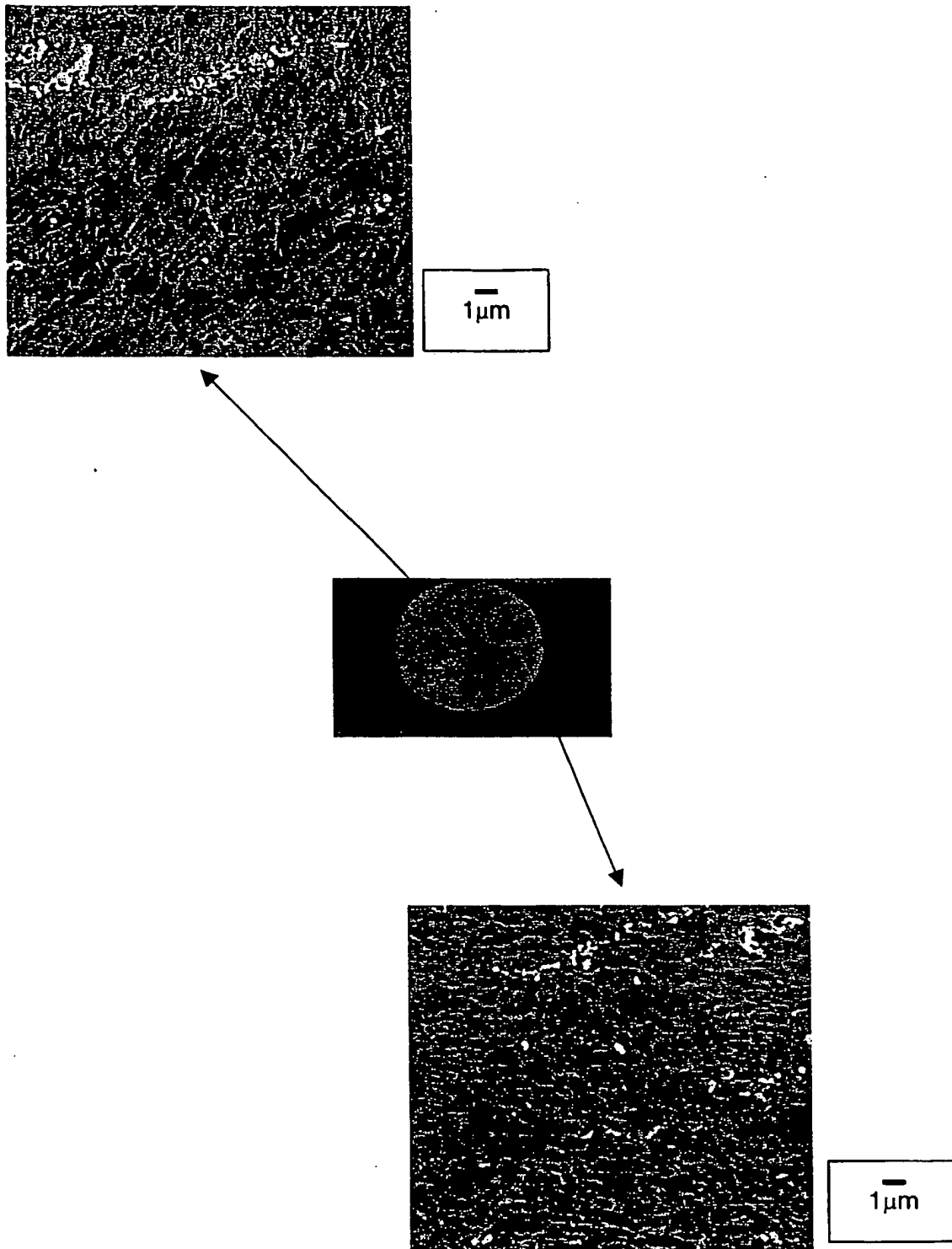


Fig.16

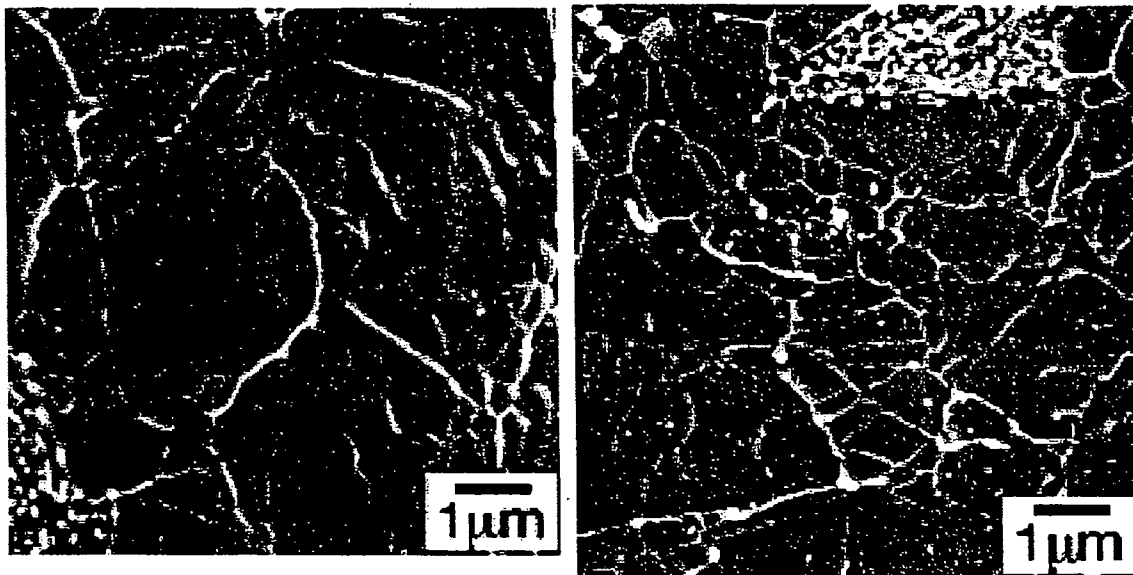
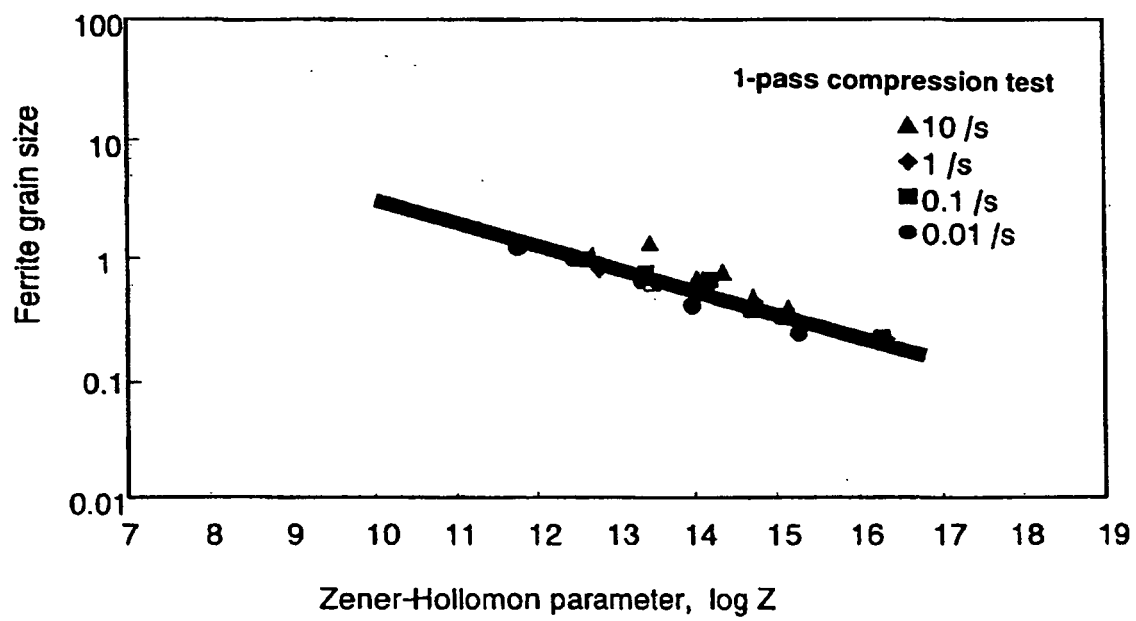


Fig.17



REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- JP 2000309850 A [0006]
- JP 2001240912 A [0006]

Non-patent literature cited in the description

- **Keizaburo Harumi et al.** Introduction to finite element method. Kyoritsu Publishing, 15 March 1990 [0027]